CHE 320: Experimental Analytical Chemistry

Spring Semester 2015 TR 9:40-10:55
David N. Blauch Tel. (704) 894-2308 dablauch@davidson.edu Office: 107 Martin

Date	Lecture Topics	Reviews
Jan 13	Course Introduction, Error Analysis, Method Validation	
Jan 15	Fundamentals of Spectrometry (**Ch. 6**) electromagnetic radiation, absorption processes	
Jan 20	Components of Spectrometers (**Ch. 7**) spectrometer design, sample preparation	
Jan 22	light sources, monochromators (prisms and diffraction gratings)	
Jan 27	detectors (photomultiplier tubes, photodiode-arrays, CCD)	
Jan 29	Molecular Spectrometry (**Ch. 13, 14**) electronic transitions, selection rules	
Feb 3	Beer's Law, deviations from Beer's Law, applications	
Feb 5	Infrared Spectrometry (**Ch. 16, 17**) rotational-vibrational spectra, selection rules	
Feb 10	Fourier transforms and FT-IR instruments	
Feb 12	Molecular Luminescence (**Ch. 15**) excitation and emission processes	
Feb 17	Raman Spectrometry (**Ch. 18**) inelastic scattering, instrumentation, applications	1st Review out
Feb 19	Atomic Spectrometry (**Ch. 8, 9, 10**) atomization processes, interferences	
Feb 24	flame atomization, graphite furnace, inductively coupled plasma emission	
Feb 26	Mass Spectrometry (**Ch. 11, 20**) isotope peaks, EI and CI ion sources	**1st Review due**
Mar 3	**Spring Break**	
Mar 5	**Spring Break**	
Mar 10	fragmentation reactions (cleavage reactions, rearrangements), interpreting spectra	
Mar 12	instrumentation: magnetic sector, double-focusing, FT-ICR	
Mar 17	instrumentation: time-of-flight, quadrupole, ion-trap, orbitrap	
Mar 19	ion sources: FAB, MALDI, ESI, ICP-MS	2nd Review out
Mar 24	**ACS National Meeting**	
Mar 26	Chromatographic Separations (**Ch. 26**) chromatograph design, flow rate, retention time	
Mar 31	column efficiency and plate theory, kinetic model, optimizing resolution	
Apr 2	Gas Chromatography (**Ch. 27**) stationary phases, retention indices	**2nd Review due**
Apr 7	**Easter Break**	
Apr 9	instrumentation: injectors, detectors, solid-phase micro-extraction	
Apr 14	Liquid Chromatography (**Ch. 28**) stationary and mobile phases, separation techniques	
Apr 16	instrumentation: injectors, detection methods	
Apr 21	GC-MS and LC-MS, tandem mass spectroscopy	3rd Review out
Apr 23	Nuclear Magnetic Resonance Spectrometry (**Ch. 19**) magnetic moment, precession	
Apr 28	Bloch equations, free-induction decay, spectrum, quadrature detection	
Apr 30	pulse sequences, inversion recovery, spin echo, magnetic resonance imaging	**3rd Review due**
May 5	Course Evaluations	

TEXT

Douglas A. Skoog, F. James Holler, and Stanley R. Crouch, *Principles of Instrumental Analysis*, 6th ed., Thomson Brooks/Cole: Belmont, CA, 2007. ISBN: 978-0-495-01201-6

COURSE RESOURCES

http://www.chm.davidson.edu/dablauch/che320/

ATTENDANCE

Attendance at all lecture and laboratory sessions is mandatory. Failure to attend lecture or laboratory sessions will result in deductions from the class participation grade. All missed laboratory work must be completed before the exam period.

GRADE SCALE

		A	93 - 100%	A-	90 - 93%
B+	87 - 90%	B	83 - 87%	B-	80 - 83%
C+	77 - 80%	C	73 - 77%	C-	70 - 73%
D+	67 - 70%	D	60 - 67%		
F	< 60%				

COURSE GRADE

	Points
First Review	100
Distributed Feb 17; due Feb 27 at 9:40 am.	
Topics: Spectrometry (UV-Vis, IR, Luminescence, Raman) (Chapters 6, 7, 13-18)	
Second Review	100
Distributed Mar 19; due Apr 3 at 9:40 am.	
Topics: Atomic Spectrometry, Mass Spectrometry (Chapters 8-11, 20)	
Third Review	100
Distributed Apr 21; due Apr 30 at 9:40 am.	
Topics: Chromatography (GC, LC) (Chapters 26-28)	
Final Exam	100
Exam center will be used	
Topics: Cumulative, with emphasis on NMR Spectrometry (Ch. 19)	
Laboratory Reports	
Analysis of Components of Soft Drinks by Liquid Chromatography	100
Oral report, PowerPoint file and Data sheet	
Simultaneous Determination of Multiple Analytes by Visible Spectroscopy	100
Excel file (raw data and results)	
Analysis of Edible Fats and Oils by ^{1}H NMR and IR Spectrometry	100
Poster (PowerPoint file) (Do not print the poster!) and spectra (with integrations)	
Determination of *o*-Xylene in Gasoline by GC-MS	100
PowerPoint file	
Spike-Recovery Analysis for the Determination of Phenol in Wasterwater	100
Formal Report (Word)	
Laboratory Notebook	50
Class Participation and Laboratory Performance	50
Total:	1000

LABORATORY PROGRAM

Laboratory Sections meet Monday 1:30 to 5:15 or Tuesday 1:00 to 4:45.

All students are required to wear safety glasses during laboratory periods.

The guidelines for each experiment are posted on the course web page. Be sure to download and read these guidelines **before** arriving for the laboratory session. Read any relevant notes or other background material. Have a clear idea of the experimental work you will be performing. If any issues are unclear, ask questions as soon as you arrive for lab. For some experiments, **students must prepare or obtain items before arriving for the lab session**.

After completing an experiment, students are responsible for cleaning their work space, disposing of any waste solutions (consult the instructor for disposal procedures), and washing all glassware used in the experiment. The work space and all glassware must be clean and ready for use by the next group at the end of the laboratory period. Students are also responsible for keeping the work area around instruments clean. All samples must be removed from instruments when measurements are complete.

Laboratory Notebooks

Each student is required to maintain a laboratory notebook, which will be graded at the end of the year. The laboratory notebook should comply with the following guidelines.

- Use a bound notebook (no loose pages) and make all entries in indelible ink.
- All pages should be numbered and records should be entered in chronological order (do not skip pages).
- The date and title should appear at the top of **each** page.
- Maintain a table of contents at the beginning of the notebook.
- Each experiment should contain the following entries:

Introduction	Describe the objectives and experimental approach of the experiment. You may include background information, physical or chemical properties, and/or experimental plans. This section must be completed **before** arriving for the laboratory period.
Experimental	**During the laboratory period**, record your experimental observations **directly** into the notebook. Include sketches of unusual experimental equipment. Indicate the name and location of any computer files containing experimental data, and indicate the nature of the information in the file. Each student should enter all information into his or her own notebook.
Results	Indicate the equations employed to analyze the experimental data, and summarize the final results of the experiment.

Laboratory Reports

Due dates for reports are indicated on the laboratory schedule. Late work will be penalized 3% per day (including weekends). Work that is more than two weeks late will not be accepted and will receive zero points. No work will be accepted after May 6, 2015.

Students may discuss the experiment, data, and data analysis with other students, but each student must perform his/her own calculations, prepare his/her own graphs, tables, and Excel files, and write his/her own reports. Students may work together to perform lengthy computer data entry. Students may **not** copy experimental data from other groups or any part of another individual's report. **Students may not look at another student's report**. Reference to the laboratory reports of other students is not permitted except as required by the instructor.

Revised Laboratory Schedule

Dates	Experiment			
	Group A	**Group B**	**Group C**	**Group D**
Jan 12/13	**No Lab**			
Jan 19/20	**No Lab** (MLK, Jr)			
Jan 26/27	HPLC (Oral Report Feb 16/17)	GC-FID (Report due Feb 20)	Vis (Report due Feb 20)	NMR/IR (Report due Feb 20)
Feb 2/3				
Feb 9/10	Vis (Report due Mar 13)	HPLC (Oral Report Mar 9/10)	NMR/IR (Report due Mar 13)	GC-FID (Report due Mar 13)
Feb 16/17				
Feb 23/24	NMR/IR (Report due Apr 3)	Vis (Report due Apr 3)	GC-FID (Report due Apr 3)	HPLC (Oral Report Mar 30/31)
Mar 2/3 **No Lab** (Spring Break)				
Mar 9/10				
Mar 16/17	GC-FID (Report due Apr 24)	NMR/IR (Report due Apr 24)	HPLC (Oral Report Apr 20/21)	Vis (Report due Apr 24)
Mar 23/24 **No Lab** (ACS Meeting)				
Mar 30/31				
Apr 6/7	**No Lab (Easter)**			
Apr 13/14	GC-MS (Report due May 6)	No Lab	GC-MS (Report due May 6)	No Lab
Apr 20/21	No Lab	GC-MS (Report due May 6)	No Lab	GC-MS (Report due May 6)
Apr 27/28	Lab Clean-Up *****			

Laboratory reports are due at 5:00 pm on the date indicated on the schedule. HPLC oral reports are scheduled during the second week of the Visible spectroscopy experiment (schedule to be posted).

***** Laboratory clean-up is mandatory for all students. Please arrive promptly at the beginning of the laboratory period.